

Functional Guidelines for Evaluating Organic Analysis

CASE No.: 42155
LABORATORY: DATAC
ANALYSIS: PESTS

SDG No.: B7FZ0
SITE: RIVERSIDE AVENUE SITE

DATA ASSESSMENT

The current SOP HW-36 (Revision 1) August 2007, USEPA Region II Data Validation SOP for Statement of Work SOM01.2 for evaluating organic data have been applied.

Data has been reviewed according to TDF Specifications, the National Function Guidelines Report # 3 and CCS Semi-Automated Screening Results Report.

All data are valid and acceptable except those analytes rejected "R"(unusable). Due to the detection of QC problems, some analytes may have the "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detect) or "JN"(presumptive evidence for the presence of the material at an estimated value) flag. All action is detailed on the attached sheets.

The "R" flag means that the associated value is unusable. In other words, significant data bias is evident and the reported analyte concentration is unreliable.

Reviewer's

Signature: Shobitha Capil

Date: February / 07 / 2012

Peer Reviewer's

Signature: _____

Date: _____ / _____ / 2012

Verified By: _____

Date: _____ / _____ / 2012

SDG# B7FZ0

1. **HOLDING TIME:**

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

No problems found for this qualification.

2. **SURROGATES**

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

No problems found for this qualification.

3. **MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:**

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for additional qualification of data.

The relative percent difference (RPD) between the following pesticide matrix spike and matrix spike duplicate recoveries is outside criteria. Detected compounds are qualified J. Non detected compounds are not qualified.

- B7FZ0, B7FZ0MS, B7FZ0MSD
- **Heptachlor** B7FZ0, B7FZ0MS, B7FZ0MSD
- **Endrin** B7FZ0, B7FZ0MS, B7FZ0MSD
- **Dieldrin** B7FZ0, B7FZ0MS, B7FZ0MSD
- **4,4'-DDT** B7FZ0, B7FZ0MS, B7FZ0MSD

The following pesticide matrix/matrix spike duplicate samples have percent recoveries that are greater than the upper acceptance limit. Detected compounds are qualified J. Non detected compounds are not qualified.

- B7FZ0, B7FZ0MS, B7FZ0MSD
- **4,4'-DDT** B7FZ0, B7FZ0MS, B7FZ0MSD
- **Endrin** B7FZ0, B7FZ0MS, B7FZ0MSD

The following pesticide matrix/matrix spike duplicate samples have percent recoveries that are less than the lower expanded criteria limit. Detected compounds are qualified J. Non detected compounds are qualified JJ.

- B7FZ0, B7FZ0MS, B7FZ0MSD

- 4,4'-DDT B7FZ0, B7FZ0MS, B7FZ0MSD
- Dieldrin B7FZ0, B7FZ0MS, B7FZ0MSD

4. The LCS data is generated to determine the long-term precision and accuracy of the analytical method. The LCS may be used in conjunction with other QC criteria for additional qualification of data.

No problems found for this qualification.

5. **BLANK CONTAMINATION:**

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Depending on the concentration of the analyte in the blank, the analytes are qualified as non-detects, "U".

The following analytes in the sample shown were qualified "U" for these reasons:

A) **Method/Instrument blank contamination:**

The following pesticide samples have analyte concentrations reported less than the CRQL. The associated method blank concentration is less than the concentration criteria. Detected compounds are qualified U. Non detected compounds are not qualified. Reported sample concentrations have been elevated to the CRQL.

- B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ1, B7FZ2, PLCSS1
- **Endrin ketone** B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ1, B7FZ2, PLCSS1
- **beta-BHC** B7FZ0, B7FZ0MS, B7FZ0MSD, PLCSS1

The following pesticide samples have analyte concentrations reported less than the CRQL. The associated continuing instrument blank concentration is less than the concentration criteria. Detected compounds are qualified U. Non detected compounds are not qualified. Reported sample concentrations have been elevated to the CRQL.

- B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ2
- **Endrin aldehyde** B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ2

B) **Field or rinse blank contamination:**

Not applicable.

6. **CALIBRATION:**

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) **Percent Relative Standard Deviation (%RSD) and Percent Difference (%D):**

For the PESTICIDE fraction, if %RSD exceeds 20% for all analytes except alpha-BHC and delta-BHC 25%, for the two surrogates and Toxaphene 30%, qualify all associated positive results "J" and non-

detects are not qualified.

B) The Percent Difference (%D) for each of the SCP and surrogate in the PEM used for CCV must be greater than or equal to -25% and less than or equal to 25.0%. The Percent Difference (%D) between the calibration Factor (CF) for each of the SCP and surrogate in the Calibration Verification Standard (CS3) and the mean calibration factor from the initial calibration must be greater than or equal to -20% and less than or equal to 20.0%. The Percent Difference not within limits, detected associated compounds are qualified "J" and non-detected associated compounds are qualified "UJ".

The following analytes in the sample shown were qualified for %RSD and %D:

No problems found for this qualification.

7. COMPOUND IDENTIFICATION:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns and a GC/MS confirmation is required if the concentration exceeds 10ng/ml in the final sample extract.

QUALIFIED U:

The following pesticide samples have percent differences between analyte results exceeding 50% and the results are below CRQL. Detected compounds are qualified U. Non detected compounds are not qualified. Reported sample concentrations have been elevated to the CRQL.

- B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ1, B7FZ2, PBLKS1, PLCSS1
- Endosulfan sulfate B7FZ0, B7FZ0MSD, B7FZ1
- 4,4'-DDE B7FZ1, B7FZ2
- gamma-Chlordane B7FZ2
- Dieldrin B7FZ0, B7FZ2
- Endrin ketone B7FZ0, B7FZ0MS, PLCSS1
- delta-BHC B7FZ0, B7FZ1, B7FZ2, PLCSS1, B7FZ0MS, B7FZ0MSD
- Methoxychlor B7FZ0MS, B7FZ0MSD, B7FZ1, B7FZ2
- Endrin B7FZ0, B7FZ1, B7FZ2
- Endrin aldehyde B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ2
- Heptachlor epoxide B7FZ0, B7FZ0MS, B7FZ0MSD, B7FZ1, B7FZ2
- beta-BHC B7FZ0, B7FZ0MS, B7FZ0MSD

QUALIFIED J:

PDL312-The following pesticide samples have percent differences between analyte results in the range of 26-50%. Detected compounds are qualified J.

- B7FZ0MSD, B7FZ2, PLCSS1
- Heptachlor B7FZ0MSD
- Dieldrin PLCSS1
- Endrin ketone B7FZ2

The following pesticide samples have percent differences between analyte results exceeding 50% and the results are below CRQL. Using professional judgement, detected compounds are qualified J.

B7FZ0MS, B7FZ0MSD

- **Heptachlor** B7FZ0MS
- **Dieldrin** B7FZ0MS, B7FZ0MSD,

The following pesticide samples have percent differences between analyte results exceeding 100%. Using professional judgement, detected compounds are qualified J.

- B7FZ0MS, B7FZ0MSD
- **4,4'-DDE** B7FZ0MS, B7FZ0MSD
- **4,4'-DDT** B7FZ0MS, B7FZ0MSD
- **Endrin** B7FZ0MS, B7FZ0MSD

QUALIFIED R:

The following pesticide samples have percent differences between analyte results exceeding 100%. Detected compounds are qualified R.

- B7FZ0
- **4,4'-DDE** B7FZ0

8. **CONTRACT PROBLEMS NON-COMPLIANCE:**
9. **FIELD DOCUMENTATION:** No problems.
10. **OTHER PROBLEMS:**
11. **This package contains re- extractions, re-analyses or dilution runs. Upon reviewing the QA results, the following Form 1(s) are identified NOT to be used.**

None.